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Part II

Environmental Protection Agency

National Emission Standards for Hazardous Air Pollutants; Alternative Test Method 107A (Vinyl Chloride)

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 61

[AD-FRL 1643-6]

National Emission Standards for Hazardous Air Pollutants; Alternative Test Method 107A (Vinyl Chloride)

AGENCY: Environmental Protection Agency (EPA).

ACTION: Proposed rule and notice of public hearing.

SUMMARY: The proposed method would apply to the measurement of the vinyl chloride content of solvents, resin-solvent solution, polyvinyl chloride resin, resin-slurry, wet resin, and latex samples. The proposed method was derived from a test method submitted to EPA by Union Carbide. The intent of this proposed method is to provide this alternative analytical procedure because it may be preferred over Method 107 in some circumstances.

A public hearing will be held to provide interested persons an opportunity for oral presentation of data views, or arguments concerning the proposed method.

DATES: *Comments.* Comments must be received on or before April 13, 1981.

Public Hearing. A public hearing will be held on March 26, 1981 (about 30 days after proposal) beginning at 9 a.m.

Request to Speak at Hearing. Persons wishing to present oral testimony must contact EPA by March 19 (1 week before hearing).

ADDRESSES: *Comments.* Comments should be submitted (in duplicate if possible) to: Central Docket Section (A-130), Attention: Docket A-80-37, U.S. Environmental Protection Agency, 401 M Street, SW., Washington, D.C. 20460.

Public Hearing. The public hearing will be held at Emissions Measurement Laboratory Building, Page Road and Interstate 40, R.T.P. North Carolina 27711. Persons wishing to present oral testimony should notify Ms. Deanna Tilley, Standards Development Branch (MD-13), U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone number (919) 541-5421.

Docket. Docket No. A-80-37, containing material relevant to this rulemaking, is available for public inspection and copying between 8:00 a.m. and 4:00 p.m., Monday through Friday, at EPA's Central Docket Section, West Tower Lobby, Gallery 1, Waterside Mall, 401 M Street, SW., Washington, D.C. 20460. A reasonable fee may be charged for copying.

FOR FURTHER INFORMATION CONTACT: Mr. Roger T. Shigehara (MD-19), U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone number (919) 541-2237.

SUPPLEMENTARY INFORMATION: On October 21, 1976 (41 FR 46560) and on June 7, 1977 (42 FR 29005) the Environmental Protection Agency promulgated Method 107—Determination of Vinyl Chloride Content of Inprocess Wastewater Samples and Vinyl Chloride Content of Polyvinyl Chloride Resin, Slurry, Wet Cake, and Latex Samples. Since that time, Union Carbide has submitted comparative supporting data to EPA on a test method that embodies a less sophisticated, but also technically satisfactory, analytical approach. Because practical considerations would favor its use in some instances, this alternative method is being considered for adoption as an EPA method. If adopted, this alternative test method would be available to determine compliance with the national emission standard for vinyl chloride, 40 CFR, Part 61 Subpart F.

(Secs. 112, 114, and 301(a) of the Clean Air Act as amended (42 U.S.C. 7412, 7414, and 760.(a)))

Dated: February 4, 1981.

Walter C. Barber,

Acting Administrator.

It is proposed to amend 40 CFR Part 61 by adding Method 107A to Appendix B as follows:

Appendix B—Test Methods

* * * * *

Method 107A—Determination of Vinyl Chloride Content of Solvents, Resin-Solvent Solution, Polyvinyl Chloride Resin, Resin Slurry, Wet Resin, and Latex Samples[1]

Introduction

Performance of this method should not be attempted by persons unfamiliar with the operation of a gas chromatograph or by those who are unfamiliar with source sampling because knowledge beyond the scope of this presentation is required. Care must be exercised to prevent exposure of sampling personnel to vinyl chloride, a carcinogen.

1. *Applicability and Principle.*

1.1 Applicability. This is an alternative method and applies to the measurement of the vinyl chloride content of solvents, resin solvent solutions, PVC resin, wet cake slurries, latex, and fabricated resin samples. This method is not acceptable where methods from Section 304(h) of the Clean Water Act, 33 U.S.C. 1251 et seq. (the Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977) are required.

1.2 Principle. The basis for this method lies in the direct injection of a liquid sample into a chromatograph and the subsequent evaporation of all volatile material into the carrier gas stream of the chromatograph, thus permitting analysis of all volatile material including vinyl chloride.

2. *Range and Sensitivity.*

The lower limit of detection of vinyl chloride in dry PVC resin is 0.2 ppm. For resin solutions, latexes, and wet resin, this limit rises inversely as the nonvolatile (resin) content decreases.

With proper calibration the upper limit may be extended as needed.

3. *Interferences.*

The chromatograph columns and the corresponding operating parameters herein described normally provide an adequate resolution of vinyl chloride. In cases where resolution interferences are encountered, the chromatograph operator shall select the column and operating parameters best suited to his particular analysis problem, subject to the approval of the Administrator. Approval is automatic, provided that the tester produces confirming data through an adequate supplemental analytical technique, such as analysis with a different column or GC/mass spectroscopy, and has the data available for review by the Administrator.

4. *Precision and Reproducibility.*

A standard sample of latex containing 181.8 ppm vinyl chloride analyzed 10 times by the alternative method showed a standard deviation of 7.5 percent and a mean error of 0.21 percent.

A sample of vinyl chloride copolymer resin solution was analyzed 10 times by the alternative method and showed a standard deviation of 6.6 percent at a level of 35 ppm.

5. *Safety.*

Do not release vinyl chloride to the laboratory atmosphere during preparation of standards. Venting or purging with vinyl chloride monomer (VCM) air mixtures must be held to minimum. When purging is required, the vapor must be routed to outside air. Vinyl chloride, even at low-ppm levels, must never be vented inside the laboratory.

6. *Apparatus.*

6.1 Sampling. The following equipment is required:

6.1.1 Glass Bottles. 16-oz wide mouth with polyethylene-lined, screw-on tops.

[1] Mention of trade names or specific products does not constitute endorsement by the U.S. Environmental Protection Agency.

6.1.2 Adhesive Tape. To prevent loosening of bottle tops.

6.2 Sample Recovery. The following equipment is required:

6.2.1 Glass Vials. 20-ml capacity with polycone screw caps.

6.2.2 Analytical Balance. Capable of weighing to ±0.01 gram.

6.2.3 Syringe. 50-microliter size, with removable needle.

6.2.4 Fritted Glass Sparger. Fine porosity.

6.2.5 Aluminum Weighing Dishes.

6.2.6 Sample Roller or Shaker. To help dissolve sample.

6.3 Analysis. The following equipment is required:

6.3.1 Gas Chromatograph. Hewlett Packard Model 5720A or equivalent.

6.3.2 Chromatograph Column. Stainless steel, 6.1 m by 3.2 mm, packed with 20 percent Tergitol E–35 on Chromosorb W AW 60/80 mesh. The analyst may use other columns provided that the precision and accuracy of the analysis of vinyl chloride standards are not impaired and that he has available for review information confirming that there is adequate resolution of the vinyl chloride peak. (Adequate resolution is defined as an area overlap of not more than 10 percent of the vinyl chloride peak by an interferent peak. Calculation of area overlap is explained in Appendix C, Supplement A: "Determination of Adequate Chromatographic Peak Resolution.")

6.3.3 Valco Instrument Six-Port Rotary Valve. For column back flush.

6.3.4 Septa. For chromatograph injection port.

6.3.5 Injection Port Liners. For chromatograph used.

6.3.6 Regulators. For required gas cylinders.

6.3.7 Soap Film Flow Meter. Hewlett Packard No. 0101–0113 or equivalent.

6.4 Calibration. The following equipment is required:

6.4.1 Analytical Balance. Capable of weighing to ±0.0001 g.

6.4.2 Erlenmeyer Flask With Glass Stopper. 125 ml.

6.4.3 Pipets. 0.1, 0.5, 1, 5, 10, and 50 ml.

6.4.4 Volumetric Flasks. 10 and 100 ml.

7. *Reagents.*

Use only reagents that are of chromatograph grade.

7.1 Analysis. The following items are required:

7.1.1 Hydrogen Gas. Zero grade.

7.1.2 Nitrogen Gas. Zero grade.

7.1.3 Air. Zero grade.

7.1.4 Tetrahydrofuran (THF). Reagent grade.

Analyze the THF by injecting 10 microliters into the prepared gas chromatograph. Compare the THF chromatogram with that shown in Figure 107A–1. If the chromatogram is comparable to A, the THF should be sparged with pure nitrogen for approximately 2 hours using the fritted glass sparger to attempt to remove the interfering peak. Reanalyze the sparged THF to determine whether the THF is acceptable for use. If the scan is comparable to B, the THF should be acceptable for use in the analysis.

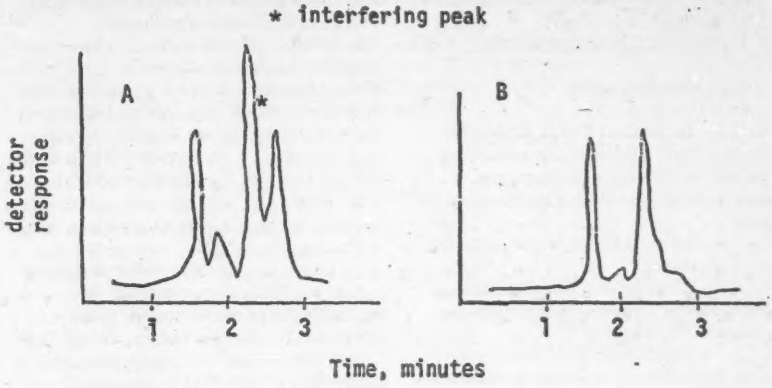

Figure 107A-1

7.1.5 N, N-Dimethylacetamide (DMAC). Spectrographic grade. For use in place in THF.

7.2 Calibration. The following item is required:

7.2.1 Vinyl Chloride 99.9 Percent. Ideal Gas Products lecture bottle, or equivalent. For preparation of standard solutions.

8. *Procedure.*

8.1 Sampling. Allow the liquid or dried resin to flow from a tap on the tank, silo, or pipeline until the tap has been purged. Fill a wide-mouth pint bottle, and immediately tightly cap the bottle. Place an identifying label on each bottle and record the date, time, sample location, and material.

8.2 Sample Treatment. Samples must be run within 24 hours.

8.2.1 Resin Samples. Weight 9.00 ± 0.01 g of THF or DMAC in a tared 20-ml vial. Add 1.00 ± 0.01 g or resin to the tared vial containing the THF or DMAC. Close the vial tightly with the screw cap, and shake or otherwise agitate the vial until complete solution of the resin is obtained. Shaking may require several minutes to several hours, depending on the nature of the resin.

8.2.2 Suspension Resin Slurry and Wet Resin Samples. Slurry must be filtered using a small Buchner funnel with vacuum to yield a wet resin sample. The filtering process must be continued only as long as a steady stream of water is exiting from the funnel. Excessive filtration time could result in some loss of VCM. The wet resin sample is weighed into a tared 20-ml vial with THF or DMAC as described earlier for resin samples (8.2.1) and treated the same as the resin sample. A sample of the wet resin is used to determine total solids as required for calculating the RVCM (Section 8.3.4).

8.2.3 Latex and Resin Solvent Solutions. Samples must be thoroughly mixed. Weigh 1.00 ± 0.01 g of the latex or resin-solvent solution into a 20-ml vial containing 9.00 ± 0.01 g of THF or DMAC as for the resin samples (8.2.1). Cap and shake until complete solution is obtained. Determine the total solids of the latex or resin solution sample (Section 8.3.4).

8.2.4 Solvents and Non-viscous Liquid Samples. No preparation of these samples is required. The neat samples are injected directly into the gas chromatograph.

8.3 Analysis.

8.3.1 Preparation of Gas Chromatograph. Install the chromatographic column, and condition overnight at 70°C. Do not connect the exit end of the column to the detector while conditioning.

8.3.1.1 Flow Rate Adjustments. Adjust the flow rates as follows:

a. Nitrogen Carrier Gas. Set regulator on cylinder to read 60 psig. Set column flow controller on the chromatograph using the soap film flow meter to yield a flow rate of 40 cc/min.

b. Burner Air Supply. Set regulator on the cylinder at 40 psig. Set regulator on the chromatograph to supply air to the burner to yield a flow rate of 250 to 300 cc/min using the flow meter.

c. Hydrogen. Set regulator on cylinder to read 60 psig. Set regulator on the chromatograph to supply 30 to 40 cc/min using the flow meter. Optimize hydrogen flow to yield the most sensitive detector response without extinguishing the

flame. Check flow with flow meter and record this flow.

d. Nitrogen Back Flush Gas. Set regulator on the chromatograph using the soap film flow meter to yield a flow rate of 40 cc/min.

8.3.1.2 Temperature Adjustments. Set temperature as follows:

a. Oven (chromatographic column) at 70°C.

b. Injection Port at 100°C.

c. Detector at 300°C.

8.3.1.3 Ignition of Flame Ionization Detector. Ignite the detector according to the manufacturer's instructions. Allow system to stabilize approximately 1 hour.

8.3.1.4 Recorder. Set pen at zero and start chart drive.

8.3.1.5 Attenuation. Set attenuation to yield desired peak height depending on sample VCM content.

8.3.2. Chromatographic Analyses.

a. Sample Injection. Remove needle from 50-microliter syringe. Open sample vial and draw 50-microliters of THF or DMAC sample recovery solution into the syringe. Recap sample vial. Attach needle to the syringe and while holding the syringe vertically (needle uppermost), eject 40 microliters into an absorbent tissue. Wipe needle with tissue. Now inject 10 microliters into chromatograph system. Repeat the injection until two consecutive values for the height of the vinyl chloride peak do not vary more than 5 percent. Use the average value for these two peak heights to compute the sample concentration.

b. Back Flush. After 4 minutes has elapsed after sample injection, actuate the back flush valve to purge the first 4 feet of the chromatographic column of solvent and other high boilers.

c. Sample Data. Record on the chromatograph strip chart the data from the sample label.

d. Elution Time. Vinyl chloride elutes at 2.8 minutes. Acetaldehyde elutes at 3.7 minutes. Analysis is considered complete when chart pen becomes stable. After 5 minutes, reset back flush valve and inject next sample.

8.3.3 Chromatograph Servicing.

a. Septum. Replace after five sample injections.

b. Sample Port Liner. Replace the sample port liner with a clean spare after five sample injections.

c. Chromatograph Shut Down. If the chromatograph has been shut down overnight, rerun one or more samples from the preceding day to test stability and precision prior to starting on the current day's work.

8.3.4 Determination of Total Solids (T.S.). For wet resin, resin solution, and PVC latex samples, determine the T.S. for each sample by accurately weighing approximately 3 to 5 grams of sample into a tared aluminum pan. The initial procedure is as follows:

a. Where water is the major volatile component: Tare the weighing dish, and add 3 to 5 grams of sample to the dish. Weigh to the nearest milligram.

b. Where volatile solvent is the major volatile component: Transfer a portion of the sample to a 20-ml screw cap vial and cap immediately. Weigh the vial to the nearest milligram. Uncap the vial and transfer a 3- to 5-gram portion of the sample to a tared aluminum weighing dish. Recap the vial and reweigh to the nearest milligram. The vial weight loss is the sample weight.

To continue, now place the weighing pan in a 130°C oven for 1 hour. Remove the dish and allow to cool to room temperature in a desiccator. Weigh the pan to the nearest 0.1 mg. Total solids is the weight of material in the aluminum pan after heating divided by the net weight of sample added to the pan originally times 100.

9. *Calibration of the Chromatograph.*

9.1 Preparation of Standards. Prepare a 1 percent by weight (approximate) solution of vinyl chloride in THF or DMAC by bubbling vinyl chloride gas from a cylinder into a tared 125-ml glass-stoppered flask containing THF or DMAC. The weight of vinyl chloride to be added should be calculated prior to this operation, i.e., 1 percent of the weight of THF or DMAC contained in the tared flask. This must be carried out in a laboratory hood. Adjust the vinyl chloride flow from the cylinder so that the vinyl chloride dissolves essentially completely in the THF or DMAC and is not blown to the atmosphere. Take particular care not to volatize any of the solution. Stopper the flask and swirl the solution to effect complete mixing. Weigh the stoppered flask to nearest 0.1 mg to determine the exact amount of vinyl chloride added.

Pipet 10 ml of the approximately 1 percent solution into a 100-ml glass-stoppered volumetric flask, and add THF or DMAC to fill to the mark. Cap the flask and invert 10 to 20 times. This solution contains approximately 1,000 ppm by weight of vinyl chloride (note the exact concentration).

Pipet 50–, 10–, 5–, 1–, 0.5–, and 0.1-ml aliquots of the approximately 1,000 ppm solution into 100 ml glass stoppered volumetric flasks. Dilute to the mark with THF or DMAC, cap the flasks and invert each 10 to 20 times. These solutions contain approximately 500, 100, 50, 10, 5, and 1 ppm vinyl chloride. Note the exact concentration of each one. These standards are to be kept under refrigeration in stoppered bottles, and must be renewed every 3 months.

9.2 Preparation of Chromatograph Calibration Curve.

Obtain the gas chromatograph for each of the six final solutions prepared in Section 9.1 by using the procedure in Section 8.3.2. Prepare a chart plotting peak height obtained from the chromatogram of each solution versus the known concentration. Draw a straight line through the points derived by the least squares method.

10. *Calculations.*

10.1 Response Factor. From the calibration curve described in Section 9.2, select the value of C_c that corresponds to H_c for each sample. Compute the response factor, R_f, for each sample as follows:

$$R_f = \frac{C_c}{H_c} \qquad \text{Eq. 107A-1}$$

10.2 Residual vinyl chloride monomer concentration (C_{rvc}) or vinyl chloride monomer concentration in resin:

$$C_{rvc} = 10 H_s R_f \qquad \text{Eq. 107A-2}$$

Where:

H_s = Peak height of sample, mm.

R_f = Chromatograph response factor.

10.3 Samples containing volatile material, i.e., resin solutions, wet resin, and latexes:

$$C_{rvc} = \frac{H_s R_f (1{,}000)}{T.S.} \qquad \text{Eq. 107A-3}$$

10.4 Samples of solvents and inprocess waste water:

$$C_{vc} = \frac{H_s R_f}{0.888} \qquad \text{Eq. 107A-4}$$

Where:

0.888 = Specific gravity of THF.

11. *Bibliography.*

1. Communication from R.N. Wheeler, Jr.; Union Carbide Corporation. Part 61 National Emissions Standards for Hazardous Air Pollutants Appendix B, Method 107—Alternate Method, September 19, 1977.

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AGENCY PUBLICATION ON ASSIGNED DAYS OF THE WEEK

The following agencies have agreed to publish all documents on two assigned days of the week (Monday/Thursday or Tuesday/Friday). This is a voluntary program. (See OFR NOTICE 41 FR 32914, August 6, 1976.)

Monday	Tuesday	Wednesday	Thursday	Friday
DOT/SECRETARY	USDA/ASCS		DOT/SECRETARY	USDA/ASCS
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DOT/FRA	MSPB/OPM		DOT/FRA	MSPB/OPM
DOT/NHTSA	LABOR		DOT/NHTSA	LABOR
DOT/RSPA	HHS/FDA		DOT/RSPA	HHS/FDA
DOT/SLSDC			DOT/SLSDC	
DOT/UMTA			DOT/UMTA	
CSA			CSA	

Documents normally scheduled for publication on a day that will be a Federal holiday will be published the next work day following the holiday.
Comments on this program are still invited.
Comments should be submitted to the Day-of-the-Week Program Coordinator. Office of the Federal Register, National Archives and Records Service, General Services Administration, Washington, D.C. 20408

NOTE: As of September 2, 1980, documents from the Animal and Plant Health Inspection Service, Department of Agriculture, will no longer be assigned to the Tuesday/Friday publication schedule.

List of Public Laws

Note: No public bills which have become law were received by the Office of the Federal Register for inclusion in today's **List of Public Laws.**

[Last Listing February 11, 1981]

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